

A Comparative Fiber Morphological Analysis of Major Agricultural Residues (Used or Investigated) as Feedstock in the Pulp and Paper Industry

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The suitability of major agricultural residues were assessed as papermaking feedstocks. All the examined agricultural residues were assumed as potential candidates for substituting hardwood fibers in mixed pulp blends from a fiber morphological perspective. Wheat, barley, rice, rapeseed, maize, sunflower, sugarcane bagasse, coconut husk, and two genotypes of miscanthus grass underwent identical maceration. The fiber length, fiber width, cell wall thickness, and lumen diameter were measured to calculate the slenderness ratio, flexibility coefficient, and Runkel ratio. The average fiber length ranged from 0.50 mm ± 0.32 mm (MG-S-02-V) to 1.15 mm ± 0.58 mm (sugarcane bagasse). The fiber width ranged from 10.77 μm ± 3.28 μm (rice straw) to 22.99 μm ± 5.20 μm (sunflower stalk). The lumen diameter ranged from 4.52 μm ± 2.52 μm (rice straw) to 13.23 μm ± 4.87 μm (sunflower stalk). The cell wall thickness ranged from 3.02 μm ± 0.95 μm (rice straw) to 4.80 μm ± 1.48 μm (sunflower stalk). The slenderness ratio, flexibility coefficient, and Runkel ratio values ranged between 28.08 to 58.11, 37.97 to 60.8, and 0.62 to 1.68, respectively. Wheat, maize, rapeseed, sugarcane bagasse, and coconut husk were found to be appropriate residue sources for papermaking feedstocks.

Keywords: Agricultural waste biomass; Non-wood fibers; Papermaking potential; Morphological indices

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INTRODUCTION

Although the primary global trend is to exploit agricultural residues (AgRs) as feedstock materials in bioenergy or biorefinery fields, not all residues are suitable for such purposes (Lal 2005). For instance, while maize and sugarcane crop residues appear as promising raw materials for bioethanol or biogas production, contrariwise, rice and wheat (44% of the total global agricultural residues production), only play a minor role in the production of biofuels (Cherubin *et al.* 2018). Moreover, second-generation biofuels, based on lignocellulosic by-products and energy (perennial herbaceous) crops, are not expected to become economically viable or commercially available in the forthcoming years; thus, second-gen biofuels were not able to contribute to reaching the 20% renewable energy consumption EU-targets by 2020 (Elbersen *et al.* 2012).

The calculated technical potential production from the eight major crops in the world, *i.e.*, wheat, maize, rice, soybean, barley, rapeseed (or canola), sugarcane, and sugar

beet, is approximately 3.3 Gton yr⁻¹ (fresh weight), as was determined by Bentsen and Felby (2010). It was also demonstrated that the Asian continent presented the largest share of crop residues production (47% of the total share), followed by the USA (29%), Europe (16%), Africa (6%), and Oceania (2%) (Cherubin *et al.* 2018). Bentsen *et al.* (2014) developed a model and pointed out that maize, rice, and wheat residues were accounting for more than three-quarters of the total production in 227 countries around the world. Likewise, Camia *et al.* (2018) and García-Condado *et al.* (2019) found that wheat, grain maize, rapeseed, barley, and sunflower residues, together constituted more than 80% of the total EU-28 residual production by using empirical models. In Asia, China and India are viewed as the two primary countries in terms of residue biomass availability, primarily in the form of wheat, maize, and rice straw in China, and sugarcane bagasse, wheat, and rice straw in India (Gregg and Smith 2010; Jiang *et al.* 2012; Hiloidhari *et al.* 2014; Chen 2016). In the USA, potential farmgate supplies of primary crop residues, *e.g.*, maize stover, wheat, barley, oats, and sorghum straw, and energy crops, *e.g.*, switchgrass, miscanthus, and biomass sorghum, were estimated to reach up to 214 Mt of crop residues and 729 Mt of herbaceous energy crops by the year 2040 by using the POLYSYS simulation assumption model (Langholtz *et al.* 2016).

For the fulfilment of the oriented global targets, set within the strategies of a sustainable and resource efficient based circular economy, it is critical to utilize alternative sources of fibrous, lignocellulosic biomass, *e.g.*, non-wood plants, as raw materials for paper and paperboard manufacturing (Przybysz *et al.* 2018; Sharma *et al.* 2018; Jeetah and Jaffur 2021). The cell types present in non-woody plants, including AgRs, are more heterogeneous than the cell types present in woody plants. The basic structure of a non-wood plant consists of vascular bundles and parenchyma tissue and contains many types of cells with a wide distribution of cellular dimensions. Furthermore, the type and size of fibers and vessel cells greatly vary within a single plant and between species, since in grasses the same type of cell may originate in different tissues and organs of the plant, which either positively or negatively influence the pulp and paper properties (Ilvessalo Pfäffli 1995; Rousu *et al.* 2013). The length of short fibers, a low bulk density, a high fines content, and a high amounts of parenchyma cells and mineral substances are a few of the most important inherent features of these non-wood plant sources (Sridach 2010). Additionally, AgRs contain lower proportions of lignin compared to wood-based sources, which is beneficial during the bleaching stage of pulp production (Kaur *et al.* 2019).

Currently, a major portion of non-wood fibers have already been used for papermaking for a long time, especially in the developing countries of Asia, Africa, and Latin America, which may feature a shortage of wood raw materials (Reddy *et al.* 2014). Conventional pulping processes, *e.g.*, soda, soda-antraquinone, and kraft, are already used for non-wood pulping, yet alternative pulping processes for non-wood pulping are more desirable. During the last years, several promising approaches in all fields of established papermaking procedures have been investigated at a laboratory or pilot scale, to overcome the limitations and challenges of non-wood pulping. These methods have constituted non-wood plant fibers as a reasonable candidate to replace wood fibers, especially replacing hardwood as the pulp feedstock (El-Sayed *et al.* 2020; Ferdous *et al.* 2020; Sharma *et al.* 2020a; Jahan *et al.* 2021).

Various non-wood plant agricultural sources have already been considered as potential pulp and paper feedstocks in the past (Ogbonnaya *et al.* 1997; Gonzalo *et al.* 2017; Saeed *et al.* 2017a; Gülsoy and Şimşir 2018; Lavrič *et al.* 2018). In addition, energy crops, and their residues, including *Miscanthus* spp. have also been considered as potential

feedstocks for paper pulps (Cappelletto *et al.* 2000; Goel *et al.* 2000; Ai and Tschirner 2010; Albert *et al.* 2011). Yet most of the studies have been focused on the investigation of cereal/rice straws, sunflower stalks, rapeseed/canola straw, and sugarcane bagasse. de Assis *et al.* (2019) found that semi-bleached wheat straw pulps (SBWP) had intermediate FL and coarseness values, with very high fines content, which results in lower bulk and water absorbency. However, this is not a limitation of producing low quality tissue products that require an intermediate combination of water absorbency, softness, and strength and could be used to replace deinked pulp. Pulps obtained from rapeseed stalks can be used as secondary fibers, replacing recycled paper in pulp blends with virgin wood fibers (González *et al.* 2013). Jeetah *et al.* (2015) demonstrated that rice husk-bagasse pulp blends (20:80 ratio) are suitable for producing insulating boards or medium packaging cardboards for decorative purposes. According to Bates *et al.* (2020) triticale pulp could be used for specific categories of printing substrates or bagasse fibers can be used for rough papers like those that are used in packaging.

Apart from China and India, the amount of produced pulp from fiber sources other than wood is still limited globally. According to FAOSTAT (2019) data, more than 80% of non-wood pulp worldwide is produced in Asian countries since the 1980s. More particularly, in these countries, the average pulp production coming from fibers other than wood was estimated at around 14258000 metric tons from 1995 to 2018. In Europe, CEPI members, which constitute 92% of the European pulp and paper industries in terms of production, the total non-wood pulp production amount hardly reached 0.8% of the total pulp production (273000 tons) (CEPI 2020).

An overall evaluation of the papermaking potential of a raw material as pulp feedstock, besides its morphological analysis, requires the evaluation of the physical properties of the obtained paper handsheets, the optimization of pulping and bleaching conditions, and the pulping chemical recovery (recycling of pulping chemicals, utilization of black liquor) processes. (Kamoga *et al.* 2016; Jahan *et al.* 2021). Non-wood fibers have unlimited differences in terms of their physical and chemical properties, and they all cover various average fiber dimensions and a wide selection of cell types and sizes (El-Sayed *et al.* 2020).

The physical properties of any paper primarily depend on its fiber morphology, fiber-fiber bonding, pulp refining, wet pressing, and formation (Sharma *et al.* 2020b). The fiber anatomical dimensions greatly impact the quality and performance of the final paper product, since these dimensions are highly correlated with its physical strength, and printing quality (Pulkkinen *et al.* 2009; Hu *et al.* 2013; Pereira *et al.* 2016). For instance, the fiber morphological properties directly affect the runnability on the paper machine, the refining response, the pulp bonding ability, and the physical, optical, and strength properties of the paper (Gülsoy and Şimşir 2018).

The fibers length (FL) and average FL distribution of a plant are considered essential morphological features, since they have a major impact on the paper strength, paper sheet formation, and drainage. Nevertheless, FL alone is not a good predictor of paper properties (Simmonds and Hyttinen 1964; Ai and Tschirner 2010; Saeed *et al.* 2017a). The fiber width (FW) and cell wall thickness (CWT) are highly correlated with fiber flexibility and bending resistance. *Arundo donax* fibers obtained from internodes parts of the plant, presented narrower LD and less wide CWT compared to those of nodes fibers, suggesting better papermaking properties (Shatalov and Pereira 2006). Furthermore, shorter LD have a positive influence on the beating of the pulp. In contrast, thicker cell walled fibers diminish the folding endurance, the burst and tensile index, and the

synergistic effect on the tear strength of a paper (Agnihotri *et al.* 2010; Tofanica *et al.* 2011; Saeed *et al.* 2017a).

In addition, the importance of plant fiber cell dimensions and their derived values on pulp and paper mechanical strength is well documented (Ververis *et al.* 2004; Nasser *et al.* 2015). Therefore, the morphological indices derivatives, *i.e.*, the slenderness ratio (SR), flexibility coefficient (FC), and Runkel ratio (RR), obtained from geometrical measurements of the fibers, are often used as an evaluating criterion to assess the suitability of a plant-based source as feedstock in papermaking production.

Additionally, pulp refining, the mechanical treatment of fibers, is a necessary step conducted on the raw source to improve the pulp quality. Depending on the pulp source, pulp consistency, refining equipment, and intensity, refining differently affects the final morphology and characteristics of the treated fibers. A few of the changes during the internal and external fibrillation of the fibers due to refining are the shortening of the fiber length, fines formation, and fibers' straightening (Gharehkhani *et al.* 2015). Therefore, the fiber morphological parameters, *i.e.*, fiber length, fiber width, lumen diameter, and cell wall thickness, of the raw non-wood plant fibers are important quality factors influencing both the pulp and paper properties and are essential to predicting the strength properties of the produced paper grades.

Thus, an initial morphological evaluation of raw non-wood fiber dimensions is a needful assessment to take into consideration, regarding the properties of the produced pulp and paper. Nevertheless, up to now, there are contradictory findings when it comes to the previous extended literature. The objective of this study was to conduct a unified fiber morphological parameter analysis, by applying the same maceration treatment and calibration/measurement method on the raw AgRs. Finally, this article aimed at detecting the potential differences between the novel results of the authors and the existing literature, hoping to infer the most appropriate AgRs feedstock for the pulp and paper industry.

EXPERIMENTAL

Materials and Methods

Raw materials

For this study, the following 10 AgRs sources were investigated: wheat straw (*Triticum spp.*), barley straw (*Hordeum vulgare*), maize stalk (*Zea mays*), rice straw (*Oryza sativa*), sunflower stalk (*Helianthus annuus*), rapeseed (*Brassica napus* L.), and sugarcane bagasse (*Saccharum officinarum*). In addition, coconut husk (*Cocos nucifera*) fibers, and from the perspective of energy crops, two genotypes of *Miscanthus x giganteus* grass residues were examined (labelled as MG-S-O2-V and MG-S-01-P, respectively) (Fig. 1).

Wheat straw, barley straw, rapeseed straw, maize stalks, and sunflower stalks samples were collected by local farmers in the region of Győr-Moson-Sopron, Hungary. The *Miscanthus x giganteus* stalks, were provided by Energianoveny Ltd., (Lengyeltóti, Hungary). The rice straw, coconut husk coir fibers, and sugarcane bagasse were obtained from local producers in Vietnam, while their chemical treatment and measurement analysis were performed in Hungary. All samples were air-dried, chopped into 3 to 5 cm length pieces, and finally stored in sealed polyethylene bags until sample preparation.

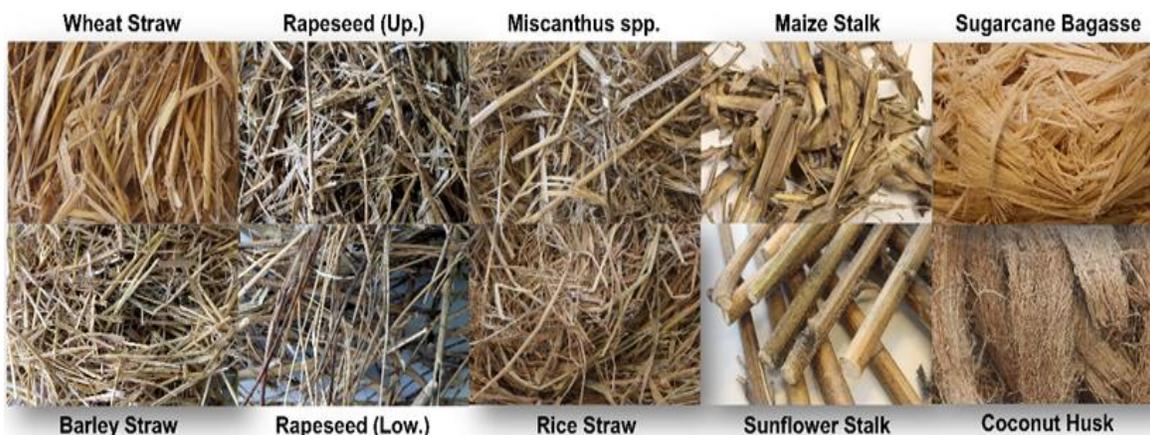


Fig. 1. Raw material of the agricultural residues (AgRs) examined in this study

Sample preparation

The non-wood AgRs were macerated according to the method performed by Danielewicz *et al.* (2018). Approximately 2 mm x 2 mm x 5 mm sticks were cut using a sharp knife. Thereafter, the sticks were placed in vials with the maceration solution, capped, and placed in a drying oven at a temperature of 60 °C for one week. The ratio of the volume of maceration solution to non-wood samples was 100 to 1 (v/w); the maceration solution was composed of one-part hydrogen peroxide (30% H₂O₂ solution), four parts of deionized water, and five parts of pure glacial acetic acid. In due course, the samples were washed and mixed with distilled water to separate the fiber bundles into individual fibers. The macerated solution samples consisted of an overall mixture of all plant parts, including the pith, nodes, and internodes.

Measurements and data processing

For the morphological analysis, optical microscope (OM) images were captured at x40 and x200 magnifications using a Nikon Eclipse 80i optical microscope (Nikon Instruments Inc., Tokyo, Japan). At least 200 fibers per AgR source were randomly measured. The number varied according to the density of each AgR solution. Image-Pro Plus software (version 6, Media Cybernetics Inc., Rockville, Maryland) was used for measuring the fiber morphological parameters. The OM images captured at x40 magnification were used for the FL measurements, while those captured at x200 were used for measuring the FW, LD, and CWT. The average values of the FL, FW, LD, and CWT parameters were calculated for each AgR source.

To assess the suitability of the AgRs as pulp feedstocks for paper production, the following three fiber morphological indices (SR, FC, and RR) were calculated according to Eqs. 1, 2, and 3,

$$SR = FL / FW \quad (1)$$

$$FC = (LD / FW) \times 100 \quad (2)$$

$$RR = 2 \times CWT / LD \quad (3)$$

(Ogbonnaya *et al.* 1997; Ververis *et al.* 2004; Albert *et al.* 2011; Mousavi *et al.* 2013; Saeed *et al.* 2017a).

RESULTS AND DISCUSSION

Fiber Dimensions Analysis

Representative OM images of the macerated fibers measured for the morphological analysis of the AgR sources are shown in Figs. 2 through 6. The OM images depict the diversity among the AgR raw materials.

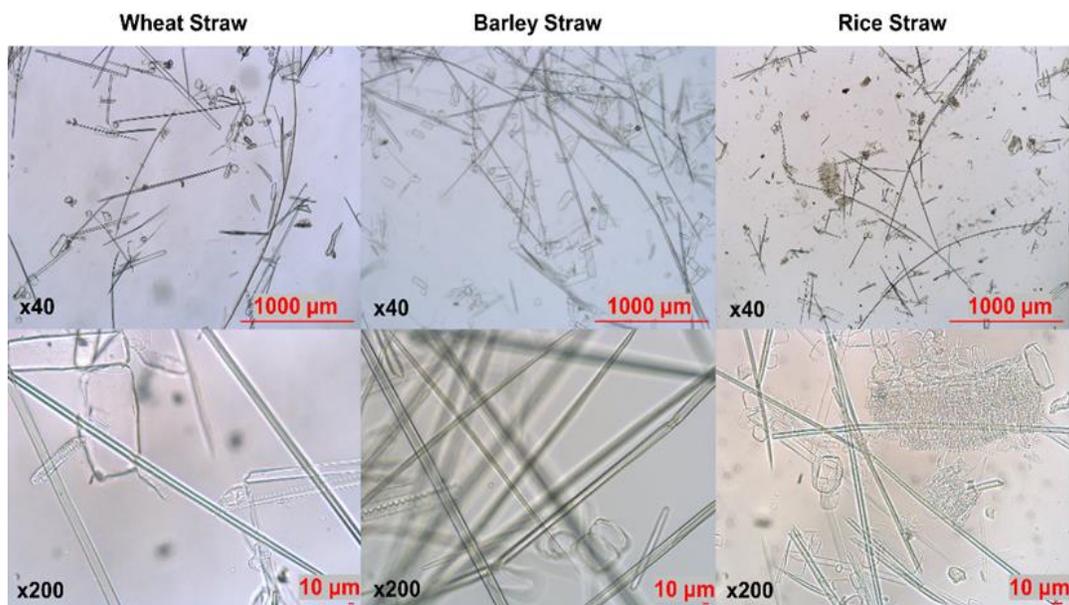


Fig. 2. Optical microscope images of the cereal straw residue fibers at ×40 and ×200 magnifications

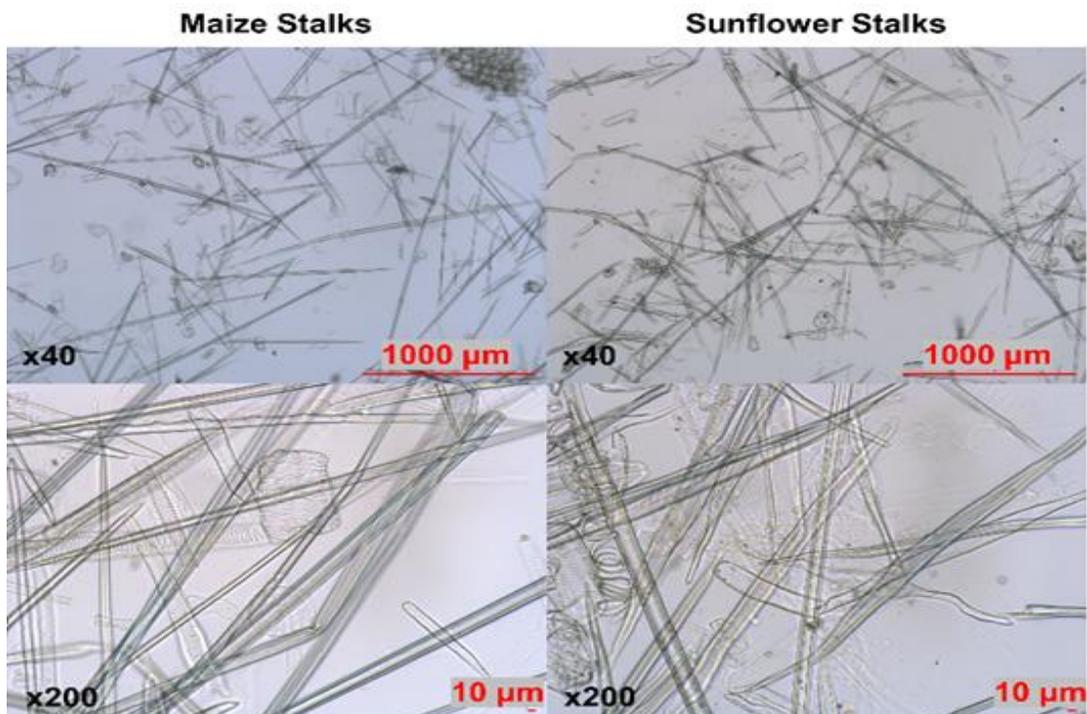


Fig. 3. Optical microscope images of the stalk residue fibers at ×40 and ×200 magnifications

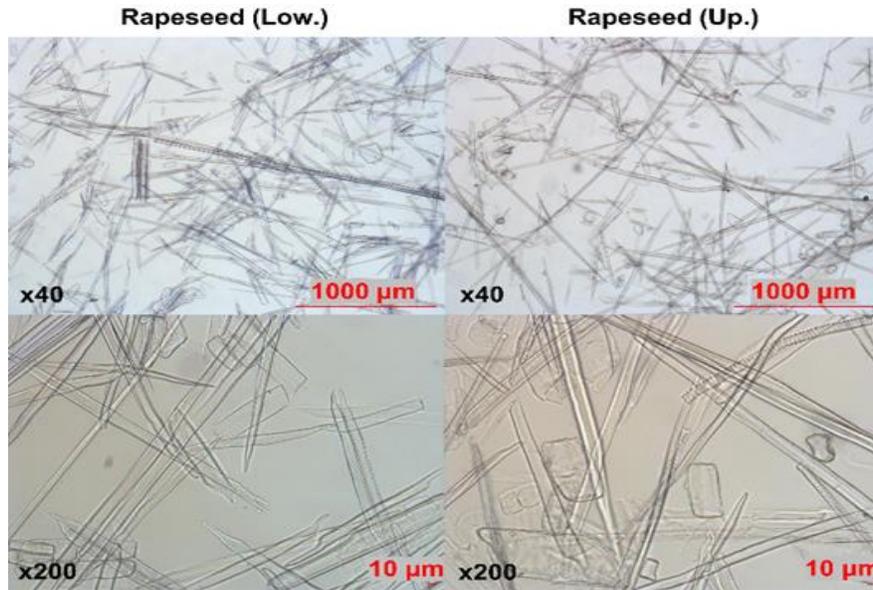


Fig. 4. Optical microscope images of the rapeseed residue fibers at x40 and x200 magnifications

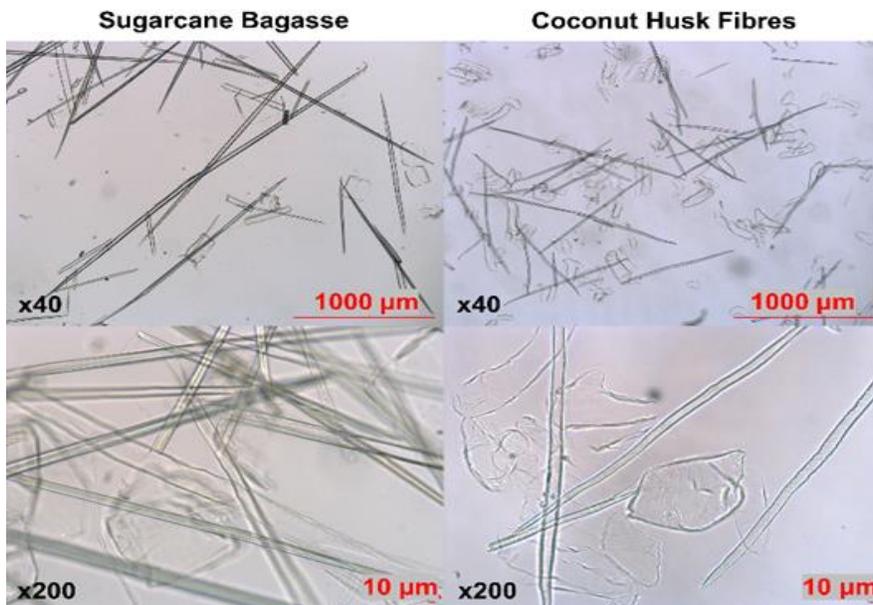


Fig. 5. Optical microscope images of the Asian originated residue fibers at x40 and x200 magnifications

The observed average FL values of the measured non-wood plant residues (Table 1) were found to be approximately within the 0.7 mm to 3.0 mm hardwood FL range (Simmonds and Hyttinen 1964; Ring and Bacon 1997). In addition, they were found to be within the FL range of the other investigated non-wood fibers obtained from vegetable AgR sources (Gonzalo *et al.* 2017; Saeed *et al.* 2017a). The sugarcane bagasse fibers presented the longest average FL ($1.15 \text{ mm} \pm 0.58 \text{ mm}$), while the *Miscanthus* MG-S-02-V fibers had the shortest ($0.50 \text{ mm} \pm 0.32 \text{ mm}$) average FL. As demonstrated by Marín *et al.* (2009) and de Assis *et al.* (2019), fibers with lengths ranging between 0.2 mm and 1.2 mm, as well as those with a length greater than 1.2 mm, are considered short and long fibers, respectively. Hardwood fibers usually are short ranging from 0.7 to 1.6 mm, with

an average fiber length of 1 mm, while softwood fibers are much longer typically ranging from 2.7 to 4.6 mm (Elmas *et al.* 2018). Therefore, the assessed AgRs can be counted as having a short FL, and short length fibers result in a denser, smoother, and more uniform paper sheet formation (Ai and Tschirner 2010).

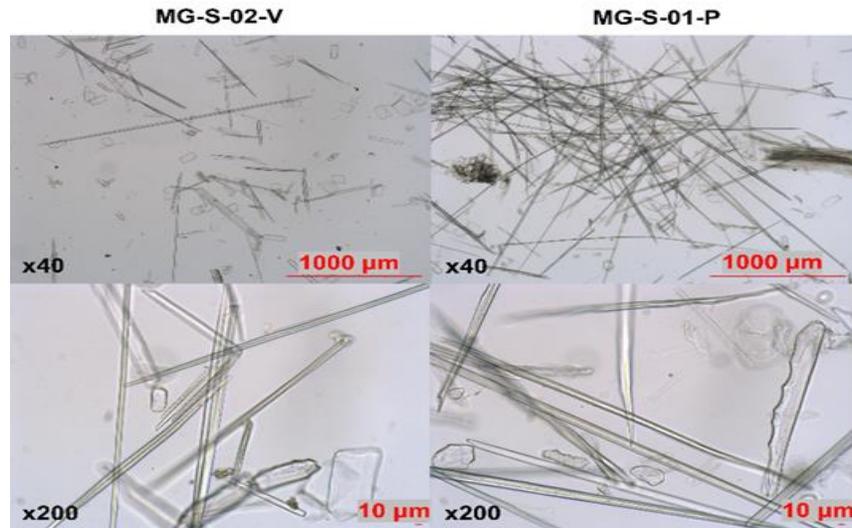


Fig. 6. Optical microscope images of the *Miscanthus* genotypes straw fibers at x40 and x200 magnifications

Table 1. Estimated Fiber Morphological Parameters of the Investigated Agricultural Residues (AgRs)

Agricultural Residue Source	FL (mm)	FW (μm)	LD (μm)	CWT (μm)
Wheat straw (<i>Triticum</i> spp.)	0.78 (± 0.44)	16.87 (± 5.46)	8.34 (± 5.12)	4.13 (± 1.40)
Barley straw (<i>Hordeum vulgare</i>)	0.67 (± 0.47)	15.26 (± 4.47)	6.97 (± 4.01)	4.07 (± 1.35)
Rice straw (<i>Oryza sativa</i> L.)	0.54 (± 0.44)	10.77 (± 3.28)	4.52 (± 2.52)	3.02 (± 0.95)
Maize stalks (<i>Zea mays</i> spp)	0.75 (± 0.31)	17.18 (± 5.65)	9.73 (± 4.69)	3.68 (± 1.13)
Sunflower stalks (<i>Helianthus annuus</i> L.)	0.64 (± 0.36)	22.99 (± 5.20)	13.23 (± 4.87)	4.80 (± 1.48)
Rapeseed up. (<i>Brassica napus</i> L.)	0.71 (± 0.53)	18.36 (± 5.58)	9.73 (± 4.57)	4.25 (± 1.27)
Rapeseed low. (<i>Brassica napus</i> L.)	0.57 (± 0.42)	18.99 (± 4.73)	11.04 (± 4.24)	3.86 (± 1.22)
Sugarcane bagasse (<i>Saccharum officinarum</i> L.)	1.15 (± 0.58)	19.86 (± 6.25)	10.25 (± 5.50)	4.50 (± 1.80)
Coconut husk/coir fibers (<i>Cocos nucifera</i>)	0.67 (± 0.27)	17.60 (± 3.12)	10.71 (± 2.73)	3.30 (± 0.69)
<i>Miscanthus x giganteus</i> Stalks MG-S-02-V	0.50 (± 0.32)	15.17 (± 4.89)	5.76 (± 3.79)	4.64 (± 1.45)
<i>Miscanthus x giganteus</i> Stalks MG-S-01-P	0.72 (± 0.43)	15.52 (± 5.06)	7.03 (± 3.98)	4.16 (± 2.21)

Table 2. Average Values of the Fiber Morphological Parameters per Agricultural Residue (AgR) Source According to Literature References

Agricultural Residue Source	FL (mm)	FW (μm)	LD (μm)	CWT (μm)	References
Wheat straw (<i>Triticum</i> spp.)	0.74 (± 0.28)	13.20 (± 2.21)	4.02 (± 1.69)	4.59 (± 1.92)	Deniz <i>et al.</i> (2004)
	0.85 (± 0.17)	9.90	6.80	1.60	Garay <i>et al.</i> (2009)
	1.18 (± 0.08)	13.60 (± 1.7)	5.68 (± 1.09)	3.96 (± 0.08)	Singh <i>et al.</i> (2011)
	1.02 (± 0.28)	11.00 (± 3.0)	8.60 (± 3.0)	1.20 (± 0.2)	Nasser <i>et al.</i> (2015)
Rice straw (<i>Oryza sativa</i> L.)	0.89	14.80	6.40	6.36	Tutus <i>et al.</i> (2004)
	0.66 (± 0.24)	4.90	1.90	1.50	Garay <i>et al.</i> (2009)
	0.83 (± 0.15)	10.89 (± 1.30)	4.57 ($\pm 0.1.37$)	3.16 (± 0.53)	Kiaei <i>et al.</i> (2011)
Maize stalks (<i>Zea mays</i> spp)	1.32	24.30	10.70	6.80	Usta <i>et al.</i> (1990)
	1.52 (± 0.49)	8.40	4.40	2.00	Garay <i>et al.</i> (2009)
	0.88 (± 0.23)	20.12 (± 3.63)	10.92 (± 3.86)	4.59 (± 0.98)	Kiaei <i>et al.</i> (2011)
Sunflower stalks (<i>Helianthus annuus</i> L.)	1.27	16.70	5.75	5.46	Khristova <i>et al.</i> (1998)
	0.96 (± 0.21)	22.84 (± 3.96)	11.12 (± 3.32)	5.85 (± 1.19)	Kiaei <i>et al.</i> (2011)
	0.96 (± 0.3)	23.70 (± 0.5)	11.90 (± 0.8)	5.90 (± 0.3)	Rudi <i>et al.</i> (2016)
Rapeseed straw (<i>Brassica napus</i> L.)	1.17	23.02	12.50	5.26	Enayati <i>et al.</i> (2009)
	1.21	28.00	11.90	7.43	Yousefi (2009)
	1.31	31.00	19.50	5.75	Hosseinpour <i>et al.</i> (2010)
	0.95 (± 0.18)	24.12 (± 6.02)	15.50 (± 5.24)	4.31 (± 1.88)	Kiaei <i>et al.</i> (2011)
	1.20 (± 0.26)	13.10 (± 3.34)	8.60 (± 2.82)	2.25 (± 0.47)	Tofanica <i>et al.</i> (2011)
	1.03	28.00	19.10	4.91	Mousavi <i>et al.</i> (2013)
Sugarcane bagasse (<i>Saccharum officinarum</i> L.)	1.70	8.40	5.20	1.60	Khristova <i>et al.</i> (2006)
	1.51 (± 0.08)	21.40 (± 1.6)	6.27 (± 0.4)	7.74 (± 0.2)	Agnihotri <i>et al.</i> (2010)
	1.59 (± 0.21)	20.96 (± 0.24)	9.72 (± 0.29)	5.64 (± 0.33)	Hemmasi <i>et al.</i> (2011)
	1.32 (± 0.30)	20.96 (± 5.03)	9.66 (± 3.32)	5.58 (± 1.54)	Kiaei <i>et al.</i> (2011)
Coconut husk/coir fibers (<i>Cocos nucifera</i>)	0.69-1.06	17.52-20.68	10.71-12.97	2.91-4.02	Dam <i>et al.</i> (2006)
	0.84 (± 0.17)	20.09 (± 3.84)	13.59 (± 3.26)	4.41 (± 1.14)	Main <i>et al.</i> (2014)
<i>Miscanthus x giganteus</i> stalks	0.97 (± 0.08)	14.2 (± 2.5)	5.90 (± 2.2)	4.10 (± 0.8)	Ververis <i>et al.</i> (2004)

Moreover, the calculated FW values in this study displayed a narrow to medium width, *i.e.*, $15.17 \mu\text{m} \pm 4.89 \mu\text{m}$ for *Miscanthus* MG-S-02-V and $22.99 \mu\text{m} \pm 5.20 \mu\text{m}$ for the sunflower stalk (apart from the narrower fibers of rice, ranging from $10.77 \mu\text{m} \pm 3.28 \mu\text{m}$). The FW ranges of these AgRs are comparable to the hardwood FW range ($18.0 \mu\text{m}$ to $30.0 \mu\text{m}$) values (Tofanica *et al.* 2011). Referring to the rest of the examined parameters, the LD ranged from $4.52 \mu\text{m} \pm 2.52 \mu\text{m}$ (rice straw) to $13.23 \mu\text{m} \pm 4.87 \mu\text{m}$ (sunflower stalk), and the CTW ranged from $3.02 \mu\text{m} \pm 0.95 \mu\text{m}$ (rice straw) to $4.80 \mu\text{m} \pm 1.48 \mu\text{m}$ (sunflower stalk).

In general, the calculated FL dimensions in this work were shown to drastically vary in many cases, with the values reported from other researchers with analogous measurements on the same non-wood species. However, the remaining fiber dimensions values of the present study, *i.e.*, the FW, LD, and CWT, were mostly found in agreement with the values reported by previous reference studies (as shown in Table 2). The notable FL differences with respect to the other studies could be explained by several reasons: (i) a variation in the total number of fibers measured to estimate the average morphological values; (ii) the crop varieties; (iii) the genotype or hybridization of the plant; (iv) software and calibration measurements; (v) the investigated parts of plants nodes, pith, internode; and (vi) the climate as well as the conditions of each collection site.

For instance, the FL increases from the base to the top for all non-wood plants, which might be a possible explanation for the observed average FL difference between the upper and lower rapeseed parts (Ververis *et al.* 2004). Furthermore, mature cereal straws and *Miscanthus* grass stems frequently do not contain pith at the internodes while retaining pith at the sections of the nodes. In other AgRs, *e.g.*, maize, sugarcane, and sunflowers, the stem remains solid, and thus the proportion of pith content is relatively higher compared to cereal straws (Ilvessalo-Pfäffli 1995). Pith mainly contains parenchyma cells, while node parts of the non-wood plants contain mostly short fibers, and parenchyma cells. Their internode parts contain longer fibers including a certain number of vessels and parenchyma cells. Therefore, the removal of the pith from the stalks is expected to increase the average FL of pulp fibers from de-pithed material (Danielewicz *et al.* 2018). The examined macerated solutions in this work contained mixed specimens of all the plant parts, compared to other studies in which the examined materials were depithed, *e.g.*, in Enayati *et al.* (2009), Hosseinpour *et al.* (2010), Kiaei *et al.* (2011), Tofanica *et al.* (2011), and Rudi *et al.* (2016). Additionally, a difference in the average FL values between the two *Miscanthus* genotypes was observed. Correspondingly, Dam *et al.* (2006) also noted slight differences in the fiber cell lengths among six coconut cultivars.

Morphological Indices and Papermaking Potential

The SR is an important parameter, that combined with the RR, helps to evaluate the morphological potential of lignocellulose fiber for paper production (Agnihotri *et al.* 2010). The SR is related to pulp digestibility, as well as the paper sheet density and tearing resistance (Ogbonnaya *et al.* 1997; Agnihotri *et al.* 2010). The higher an SR value is, the longer, thinner, and more flexible the fibers are considered to be, and subsequently, the stronger the tearing resistance of the paper sheet is expected to be (Ogbonnaya *et al.* 1997; Nasser *et al.* 2015). An optimal SR value range between 95 and 120 or between 55 to 75 for softwood and hardwood pulps, respectively, is recommended (Tofanica *et al.* 2011; Rudi *et al.* 2016; Gülsoy and Şimşir 2018). However, a non-wood plant source is considered sufficient for papermaking processes if the SR value is less than 70 and higher than 33 (Saeed *et al.* 2017a).

The FC index is related to the individual elasticity of fibers and has a positive effect on the interfibrillar bonding behaviour of fibers, and eventually on the tensile and burst strength properties. Plant fibers can be classified as highly rigid fibers (an FC less than 30) through high elastic fibers (an FC greater than 75) (Tofanica *et al.* 2011; Gülsoy and Şimşir 2018). A higher rigidity of fiber has a negative influence on the mechanical strength properties of the resulting paper and provide porous papers with lower resistance (Nasser *et al.* 2015; Saeed *et al.* 2017a).

Plant material fibers with a RR less than 1 are suggested as preferable raw material for pulp and paper production, since these fibers are more flexible, easily collapsed, and less rigid, thus forming papers with improved strength. Previous studies indicated that fibers with a RR above 1 are stiffer, less flexible, exhibit lower interfibrillar bonding, and form bulkier papers (Agnihotri *et al.* 2010; Tofanica *et al.* 2011; Nasser *et al.* 2015; Gülsoy and Şimşir 2018).

A higher average FL, combined with a higher SR, a higher FC, and lower RR values indicate higher tensile, tear, and burst strength properties. Thus, non-wood plant sources that have these morphological characteristics should be considered optimal candidates as feedstocks in the pulp and paper industries.

Table 3. Estimated Morphological Indices Values of the Investigated Agricultural Residues (AgRs)

Agricultural Residue Source	SR	FC	RR
Wheat straw (<i>Triticum</i> spp.)	46.31	49.44	0.98
Barley straw (<i>Hordeum vulgare</i>)	43.86	45.67	1.17
Rice straw (<i>Oryza sativa</i> L.)	49.90	41.97	1.34
Maize stalks (<i>Zea mays</i> spp)	43.81	56.63	0.87
Sunflower stalks (<i>Helianthus annuus</i> L.)	28.08	57.80	0.72
Rapeseed up. (<i>Brassica napus</i> L.)	38.70	53.0	0.87
Rapeseed low. (<i>Brassica napus</i> L.)	30.24	58.13	0.70
Sugarcane bagasse (<i>Saccharum officinarum</i> L.)	58.11	51.72	0.88
Coconut husk/coir fibers (<i>Cocos nucifera</i>)	38.32	60.8	0.62
<i>Miscanthus x giganteus</i> stalks MG-S-02-V	32.85	37.97	1.68
<i>Miscanthus x giganteus</i> stalks MG-S-01-P	46.21	45.30	1.18

According to the RR values classified by Tofanica *et al.* (2011), wheat straw, rapeseed straw, maize stalk, sunflower stalks, and sugarcane bagasse fibers (with a RR of 0.50 to 1.0), are suitable for paper manufacturing products and are further characterized as flexible fibers with a medium thickness cell wall. The remaining AgRs, *i.e.*, barley straw, rice straw, and miscanthus straw fibers (with a RR of 1.0 to 2.0), were found to be stiff, with thick cell walls, and small lumen fibers, and therefore are less suitable for paper products (Table 3). The morphological indices of the investigated AgRs revealed that *Miscanthus* MG-S-O2-V seems unsuitable as a pulp feedstock. However, wheat straw, maize stalks, rapeseed upper straw, sugarcane bagasse, and coconut husk were found to be suitable as a raw materials for pulp production. The remaining AgR sources can be

classified as having intermediate suitability as a non-wood pulp feedstock. In addition, barley straw, rice straw, and *Miscanthus* MG-S-01-P had a RR greater than 1, whilst sunflower stalks and rapeseed lower part straw fibers had an SR less than 33.

Table 4. Morphological Indices Values per AgRs Source According to Literature References

Agricultural Residue Source	SR	FC	RR	References
Wheat straw (<i>Triticum</i> spp.)	55.9	30.45	0.21	Deniz <i>et al.</i> (2004)
	85.76	68.69	0.47	Garay <i>et al.</i> (2009)
	86.76	41.76	1.39	Singh <i>et al.</i> (2011)
	96.4	78.2	0.28	Nasser <i>et al.</i> (2015)
Rice straw (<i>Oryza sativa</i> L.)	60.13	43.24	1.98	Tutus <i>et al.</i> (2004)
	134.70	38.77	1.58	Garay <i>et al.</i> (2009)
	76.58	41.96	1.38	Kiaei <i>et al.</i> (2011)
Maize stalks (<i>Zea mays</i> spp.)	54.32	44.03	1.27	Usta <i>et al.</i> (1990)
	180.95	52.38	0.91	Garay <i>et al.</i> (2009)
	44.08	54.27	0.84	Kiaei <i>et al.</i> (2011)
Sunflower stalks (<i>Helianthus annuus</i> L.)	76.04	34.43	1.90	Khristova <i>et al.</i> (1998)
	42.03	48.68	1.05	Kiaei <i>et al.</i> (2011)
	40.55	50.49	0.49	Rudi <i>et al.</i> (2016)
Rapeseed straw (<i>Brassica napus</i> L.)	50.83	54.30	0.84	Enayati <i>et al.</i> (2009)
	43.21	42.5	1.25	Yousefi (2009)
	42.26	62.9	0.59	Hosseinpour <i>et al.</i> (2010)
	39.59	64.26	0.55	Kiaei <i>et al.</i> (2011)
	36.8	68.21	0.51	Tofanica <i>et al.</i> (2011)
	91.0	26.00	0.58	Mousavi <i>et al.</i> (2013)
Sugarcane bagasse (<i>Saccharum officinarum</i> L.)	202.4	61.9	0.61	Khristova <i>et al.</i> (2006)
	70.56	29.30	2.46	Agnihotri <i>et al.</i> (2010)
	75.86	46.37	1.16	Hemmasi <i>et al.</i> (2011)
	62.97	46.08	1.15	Kiaei <i>et al.</i> (2011)
Coconut husk/coir fibers (<i>Cocos nucifera</i>)	45.55	61.99	0.58	Dam <i>et al.</i> (2006)
	41.81	67.64	0.65	Main <i>et al.</i> (2014)
<i>Miscanthus x giganteus</i> stalks	68.3	41.5	1.3	Ververis <i>et al.</i> (2004)

It should be noted that several morphological indices variations are documented in various literature (Table 4). The indicated differences appear to verify the necessity of an identical, integrated morphological analysis, to assess the suitability of the primary AgRs sources, which has been carried out in this work.

However, in addition to the morphological parameters of the fibers, high cellulose and low lignin contents are also essential evaluation criteria of a material planned to be used as a feedstock in pulp and paper industries and should be taken under consideration (Amode and Jeetah 2021). From a chemical point of view, cereals crop residues, *i.e.*, wheat, maize, barley, rice, and rapeseed straw, contained an average lignin content of approximately 17% to 20% and an average cellulose content of 37% to 41%. Sunflower straw exhibited intermediate lignin and cellulose compositions, *i.e.*, 25.2% lignin and 34.8% cellulose contents. *Miscanthus* grass has been reported to exhibit considerably high cellulose (46.3%) content. On the contrary, sugarcane bagasse, which in this study displayed excellent morphological results, has been reported to hold relatively low cellulose (26.8%) values (Thorenz *et al.* 2018).

CONCLUSIONS

Agricultural residues (AgRs) have already been used or examined as feedstock materials for pulps. Fiber morphological analysis is one of the most essential prerequisites to illustrate their suitability for this purpose. However, several variations are documented in the literature relevant to their morphological characteristics and indices. In this study, it was attempted to deal with such considerations. Therefore, a comparative morphological analysis under an identical maceration and measurement analysis was performed among the major AgR crops worldwide.

1. From a morphology point of view, wheat straw, maize stalks, rapeseed straw, sugarcane bagasse, and coconut husk fibers were found to present the minimum potential required morphological parameters and therefore considered as the most suitable feedstocks for papermaking processes.
2. This outcome is very encouraging since these residues primarily account for a majority of the total residual biomass availability in their classified groups, *i.e.*, cereals, oil, and sugar crops, respectively. It was also shown that the average rapeseed fiber length was slightly different, depending on the height of the plant.
3. Similarly, the average miscanthus fiber length was greatly influenced by the originated genotype.

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